(11) (A) No.   **1 214 421**

(45) ISSUED 861125

(52) CLASS   196-19

4
(51) INT. CL. C10G 1/04

(19) (CA) CANADIAN PATENT (12)

(54) Blending Tar Sands to Provide Feedstock for Hot Water
Process

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(73) Granted to Petro-Canada Exploration Inc.
Canada
Majesty (Her) the Queen in right of the Province of
Alberta as represented by the Minister of Energy and
Natural Resources (Alberta Oil Sands Equity)
Canada
PanCanadian Petroleum Limited
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Canada-Cities Service, Ltd.
Canada
Gulf Canada Limited
Canada
Alberta Energy Company Ltd.
Canada
Hudson's Bay Oil and Gas Company Limited
Canada
Petrofina Canada Inc.
Canada

(21) APPLICATION No.   442,511

(22) FILED   831202

No. OF CLAIMS   3

Canada

1 "BLENDING TAR SANDS TO PROVIDE FEEDSTOCKS
2   FOR HOT WATER PROCESS"

3   ABSTRACT OF THE DISCLOSURE

4   Two or more tar sand feeds are blended in specified pro-

5 portions in combination with a specified dosage of NaOH to provide a

6 slurry, having a free surfactant content dissolved in the aqueous phase

7 of the slurry, which yields the maximum possible primary froth

8 production from the hot water extraction process.

1
FIELD OF THE INVENTION

2   This invention relates to an improvement of the hot water

3 process for extracting bitumen from tar sand. More particularly, it

4 relates to process control, specifically control of process aid addition,

5 whereby primary bitumen froth recovery may be maximized, in spite of

6 the changing nature of the tar sand feed.

7
BACKGROUND OF THE INVENTION

8   Tar sand, also known as oil sand and bituminous sand, is

9 now well recognized as a valuable source of hydrocarbons. There are

10 presently two large plants producing synthetic crude from the tar sands

11 of the Athabasca region of Alberta. In these operations, the tar sands

12 are first mined and the bitumen is then extracted from the tar sand by a

13 process called the hot water process. The extracted bitumen is subse-

14 quently upgraded by refinery-type processing to produce the synthetic

15 crude.

16   The tar sand is a mixture of sand grains, connate water,

17 fine minerals solids of the particle size of clay minerals, and bitumen.

18 It is commonly believed that the connate water envelopes the grains of

19 sand, the fine solids are distributed in the water sheaths, and the

20 bitumen is trapped in the interstitial spaces between the water-sheathed

21 grains.

22   The hot water process is now well described in the patent

23 and technical literature.

24   In broad summary, this process comprises first conditioning

25 the tar sand, to make it amenable to flotation separation of the bitumen

26 from the solids. Conditioning involves feeding mined tar sand, hot

27 water (180°F), an alkaline process aid (usually NaOH), and steam into

28 a rotating horizontal drum wherein the ingredients are agitated together.

29 Typically, the amounts of reagents added are in the following proportions:

1	tar sand	- 3250 tons
2	hot water	- 610 tons
3	NaOH	- 4 tons (20% NaOH)

4 Enough steam is added to ensure an exit temperature of the mixture from

5 the drum of about 180°F. The residence time in the drum is typically

6 about 4 minutes.

7   During conditioning, the mined tar sand (in which the

8 bitumen, connate water and solids are tightly bound together) becomes

9 an aqueous slurry of porridge-like consistency, wherein the components

10 are in loose association.

11   The slurry leaving the drum is screened, to remove oversize

12 material, and then flooded or diluted with additional hot water. The

13 product typically comprises 7% by weight bitumen, 43% water and 50%

14 solids. Its temperature is typically 160 - 180°F.

15   The diluted slurry then is transferred to the primary

16 separation operation, where it is introduced into a large separation

17 vessel having a cylindrical upper section and conical lower section.

18 Here the slurry is retained for about 45 minutes in a quiescent con-

19 dition. Most of the sand sinks to the bottom and is discharged, to-

20 gether with some fines, water, and bitumen, through an outlet. This

21 discharge is discarded as tailings.

22   The bitumen present in the separation vessel exists in

23 the form of globules, some of which attach themselves to air bubbles

24 entrained in the slurry during conditioning. The aerated bitumen tends

25 to rise through the slurry and is recovered as a froth by a launder

26 extending around the upper lip of the separation vessel. This froth is

27 called primary froth. Typically, it comprises:

28   66.4%   bitumen

29   8.9%   solids

30   24.7%   water.

1   Not all of the bitumen becomes sufficiently aerated to rise

2   into the primary froth product. Much of this bitumen, together with

3   much of the fines, tends to collect in the mid-section of the separation

4   vessel. This aqueous mixture is termed "middlings".

5   The middlings are withdrawn from the vessel and are fed

6   into subaerated flotation cells where secondary separation is

7   practiced. Here the middlings are subjected to vigorous agitation and

8   aeration. Bitumen froth, termed "secondary froth", is produced.

9   Typically, this froth comprises:

10   23.8%   bitumen

11   17.5%   solids

12   58.7%   water.

13 It will be noted that the secondary froth is considerably

14 more contaminated with water and solids than the primary froth. One

15 seeks to minimize this contamination, as the froth stream requires

16 downstream treatment to remove solids and water, before it can be fed

17 to the upgrading process.

18 It is desirable to operate the process so that as much

19 of the bitumen as possible reports to the primary froth. The efficiency

20 with which bitumen is collected as primary froth is a measure of the

21 success with which the entire bitumen in the tar sand feed has been

22 brought to a condition amenable for spontaneous flotation. For this

23 reason, one may consider maximizing primary recovery as optimizing the

24 entire process.

25 Now, the tar sand feed to the hot water process is not

26 uniform in nature. Its properties vary in accordance with factors

27 such as bitumen content, fines content, nature of the coarse solids,

28 extent of ageing and weathering after mining, and the chemical nature

29 of the bitumen. This variation in properties of the feedstock requires

1   that the processing conditions be altered from time to time with a

2   view to maximizing primary froth recovery. Some optimizing techniques,

3   such as regulating middlings density within a preferred range or main-

4   taining the temperature within a preferred narrow range, can assist in

5   improving recovery over a narrow variation in the nature of the tar sand

6   feed. But there is a need for identification of a parameter which can be

7   monitored and used to maximize primary froth recovery over a wide range

8   of different tar sand types.

9   At this point, it is useful to review the role of the "process

10   aid", as it was understood in the past. The originator of the hot water

11   process, Dr. Karl Clark, noted that the tar sand was acidic in nature. He

12   taught the need to add an alkaline process aid, such as NaOH, to adjust

13   the pH of the drum slurry to near neutral condition, in order to improve

14   bitumen recovery in the primary separation step. Later investigators

15   taught that it was desirable to maintain a slurry pH in the range of

16   about 8 - 9, to maximize bitumen recovery.

17   More recently, Dr. Emerson Sanford, co-worker of the

18   present applicants, set forth in Canadian Patents 1,100,074 and

19   1,094,003 that the role of the NaOH was to produce surfactants in the

20   slurry by reaction with carboxylic and sulfonic acid substituents

21   present in the bitumen. He submitted that it was surfactants that

22   were needed to condition the tar sand to free the bitumen from the

23   other tar sand components and render said bitumen amenable to air

24   attachment. He further taught that the level of fines would affect

25   the surfactant requirements. It was believed the fines would adsorb

26   surfactants, thereby diminishing their availability for 'conditioning'. In

27   summary, he taught that:

1   (1) some process aid was needed for good primary recovery;

2   (2) the process aid functioned by generating surfactants

3   within the slurry, which surfactants were required

4   to maximize bitumen recovery; and

5   (3) different tar sand types, having different fines

6   contents, would require different quantities of NaOH

7   in order to achieve maximum primary froth production.

8   As mining and geological inspection of the Athabasca

9 deposit has expanded, it has become evident that there are oil sand

10 types that do not follow the relationships between recovery and process

11 aid addition that one would have anticipated.

12   One such deviation arises from the nature of the clays. It

13 now appears that clays differ in their capacity to adsorb surfactants.

14 THose that are so placed in the deposit as to be in contact with bitumen

15 can have surfaces thoroughly impregnated with hydrocarbon molecules and

16 may not have sites available for surfactant adsorption. Clays laid

17 down in later eras, and forming part of the overburden, may have

18 hydrocarbon-free surfaces,in which case they are strong surfactant

19 adsorbers. If, during mining, some of the overburden gets included

20 in the feed sent to extraction, these non-impregnated clays "poison" the

21 slurry by adsorbing surfactants. When the extraction circuit has been

22 optimized for non-adsorbing clays, the introduction of feed containing

23 overburden clays will lead to reduced recovery. Some oil sands are so

24 rich in surfactant-adsorbing clays that the power of the contained

25 bitumen to contribute surfactants to the slurry is more than offset by

26 the tendency of the clays to adsorb surfactants.

27   A second deviation from "normal" behaviour is the deterioration

28 tion of oil sand after mining. During storage, feed can age. The

29 mechanism is not understood, but the bitumen surface properties appear

30 to alter, with the result that separation from the solids and attachment

31 of air are made more difficult.

1   A third deviation arises in the case of feeds rich in bitumen.

2 Some have been found to produce such a high level of surfactants, without

3 any process aid addition, that the slurry is always in an over-conditioned

4 state. Over-conditioning results in bitumen losses from the separa-

5 tion vessel, presumably due to emulsification.

6   Trying to control the process by monitoring some property

7 of the feed is thus liable to failure because the relationships between

8 such property and recovery can be subject to abberrations.

9   A safer procedure is to use some property of the slurry,

10 once prepared, rather than of the feed, to determine the needed dosage of

11 process aid.

12   There is thus a need to identify a reliable parameter which

13 can be used to optimize NaOH addition and to determine a strategy for

14 best combining the various types of tar sand to offset their undesirable

15 qualities with respect to surfactant production and consumption.

16   SUMMARY OF THE INVENTION

17   The present invention is based in part on the discovery that,

18 for a particular circuit, there is a critical or optimum concentration of

19 free surfactant in solution in the aqueous phase of the process slurry

20 (hereinafter "C_o"), which always is required to obtain maximum recovery

21 of bitumen from tar sand in the form of primary froth.

22   The discovery was arrived at by running a number of batch

23 hot water process extractions on a single tar sand feed, while varying

24 only the amount of process aid used. When primary froth recovery was

25 plotted against concentration of free surfactant in the aqueous phase

26 of the process slurry (hereinafter "C_s"), a peak curve was obtained.

27 When this procedure was repeated for a number of different tar sands,

28 a peak curve was obtained in each case and the peaks were found to occur

29 at substantially the same C_s value. This common peak C_s value is the

30 optimum concentration C_o.

So, for a particular circuit, a single free surfactant value C_o leads to maximum primary froth recovery, regardless of the nature of the tar sand feedstock being processed.

Having made this discovery, a general process has been evolved comprising the following steps:

(1) determining, for the extraction apparatus or circuit used, a measure of C_o for one tar sand feed;

(2) then establishing C_s from time to time as different tar sand feeds are processed; and

(3) varying the process aid addition to the process as the nature of the tar sand feed changes, to thereby maintain C_s as close to C_o as possible.

The general process has now advantageously been applied in connection with feedstocks, for the hot water process, which consist of blends of two or more different tar sand feeds. If a plurality of different tar sand feeds, which satisfy criteria set forth below, are selected and combined in certain proportions, which are set forth in a range defined below, and if this blend is combined with process aid in an amount within a range defined below, then, when this mixture is used in the hot water process, two results occur:

(1) C_s is found to equate substantially with C_o ; and

(2) the primary froth recovery obtained is greater than that which would be obtained if one processed each of the tar sand feeds separately.

The use of blended feedstock and process aid as aforesaid in the hot water process is characterized by an important advantage. The invention enables certain tar sand feeds, which could not by themselves be processed at C_o condition, to be so processed.

1 More particularly, there are certain tar sand feeds which,
2 when slurried, initially are consumers of free surfactants. These tar
3 sand feeds usually have a high fines content. The fines tend to adsorb
4 the free surfactants; this is particularly the case when the fines are
5 not well impregnated with bitumen. Thus, when a tar sand of this type
6 is processed, a relatively large quantity of process aid would be required
7 to bring C_s to the C_o value. Now, there is a known limit on the amount
8 of process aid which can be used in the hot water process. This is
9 generally taken to be about 0.2 wt. %, based on the dry tar sand. If
10 this limiting amount of process aid is exceeded, the hot water process
11 is deleteriously affected by such effects as emulsification of bitumen
12 and poor froth/middlings interface. By blending these free surfactant-
13 consuming tar sand feeds with others, as described below, they can now
14 be processed at C_o condition without exceeding the 0.2 wt. % limit.
15   There are other tar sand feeds, usually high in bitumen
16 content, which, when slurried, produce such a high concentration of
17 free surfactants that they cannot be processed in conventional fashion
18 at C_o condition. These tar sand feeds, when slurried, yield a C_s
19 value which is so high that it is on the downslope of the peak curve.
20 By blending these free surfactant-producing tar sand feeds with free
21 surfactant-consuming feeds, both types of feeds can now be processed
22 at C_o condition.
23   Broadly stated, the invention is a process for extracting
24 bitumen from tar sand of varying nature using the hot water process in
25 an extraction circuit, wherein the tar sand is conditioned, by slurrying
26 it with hot water and alkaline process aid with agitation, is diluted
27 with water, and is then retained in a quiescent condition to produce
28 primary bitumen froth. The improvement comprises: selecting a first
29 tar sand feed which, when slurried, is a consumer of free surfactants
30 and a second tar sand feed which, when slurried, is a producer of free
31 surfactants; and blending said first and second tar sand feeds with

1 process aid in the conditioning step in amounts selected to yield sub-

2 stantially the optimum free surfactant condition, in the aqueous phase

3 of the process slurry for the circuit, required to yield maximum primary

4 froth recovery.

5 ## DESCRIPTION OF THE DRAWINGS

6 Figure 1 is a side view of a laboratory hot water process

7 batch extraction unit used to develop the data underlying this invention;

8 Figure 2 is a peak curve plot for various tar sand type

9 samples of primary froth recovery (%) against free surfactant concentration

10 in secondary tailings - the data was obtained by conducting extractions

11 on each feed at varying NaOH additions while holding other conditions

12 constant;

13 Figure 3 is a plot of free surfactant concentration obtained

14 in the aqueous phase of the process slurry (C_s) against dosage of NaOH

15 process aid used in the process - line A is the surfactant production

16 line obtained by treating samples of surfactant-producing tar sand A

17 with different amounts of NaOH, and line B is the surfactant production

18 line obtained by treating samples of a surfactant-consuming tar sand B -

19 the dotted horizontal line corresponds with C_o (the optimum concentration

20 of free surfactant in the aqueous phase of the process slurry for the

21 extraction unit involved, which concentration corresponds with that

22 associated with maximum primary bitumen froth recovery);

23 Figure 4 is a plot, similar to that of Figure 3, for two other

24 tar sands C and D - tar sand C differs from tar sand A in that its

25 intercept with the free surfactant concentration axis is less than C_o,

26 whereas A's is greater - tar sand C is a free surfactant-producing tar

27 sand, and tar sand D is a free surfactant-consuming tar sand;

28 Figure 5 is a plot of maximum primary froth recovery against

29 composition of blend (expressed in terms of % marine ore in the blend) -

30 this plot shows the synergism which arises with respect to primary recovery

31 when certain blends are used; and

1   Figure 6 sets forth titration curves used in the determination

2 of free surfactant concentration.

3   DESCRIPTION OF THE PREFERRED EMBODIMENT

4   The invention is a process which has evolved from laboratory

5 experimentation, using bench scale equipment and involving the measure-

6 ment of certain parameters and the treatment of the measurement data

7 acquired. It is necessary to go into this experimental background, in

8 order to comprehend the invention.

9 The Laboratory Extraction Unit

10   The laboratory batch extraction unit shown in Figure 1 was

11 used to develop the data set forth below. A detailed description of this

12 unit and its operation is given in the paper by E. C. Sanford and F. A.

13 Seyer entitled "Processability of Athabasca Tar Sand Using a Batch

14 Extraction Unit: The Role of NaOH" in the Canadian Institute of Mines

15 Bulletin, 72 , (1979), at page 164.

16   Experience has shown that the practise of the hot water

17 extraction process on tar sand in this unit gives results which closely

18 parallel those of the commercial hot water extraction process plant operated

19 by the assignees of this application.

20   The extraction unit of Figure 1 comprises a steel pot 1

21 having a hot water heating jacket 2 for temperature control. An agitator

22 3 for stirring and a sparger 4, for the introduction of air, extend into

23 the pot.

24   In the operation of the extraction unit, a charge of tar

25 sand, water and NaOH is introduced into the pot. The pot contents are

26 then heated by the jacket. Once at the desired temperature, the charge

27 is agitated and air is sparged into the slurry, to simulate the step of

28 conditioning in a drum. The air sparging is then stopped and additional

1 hot water is added, to simulate the step of flooding or dilution.

2 Agitation is continued for a few minutes, to mix the components. The

3 product is then retained in the pot under quiescent conditions, to

4 simulate primary separation or flotation. Primary bitumen froth is

5 produced during this step. This froth is skimmed off. The residual

6 mixture is then again agitated with vigorous air sparging, to simulate

7 secondary separation. Secondary froth is produced and skimmed off. The

8 material left in the pot is referred to hereafter as 'secondary

9 tailings'.

10 The determination of C_s values was arrived at using the

11 aqueous phase of this secondary tailings as the feedstock for analysis.

12 The bitumen recoveries were based on the bitumen contents and amounts of

13 the froth produced and skimmed off.

14 Free Surfactant (C$_s$) Determination

15 The invention has necessitated the utilization of a method

16 for measuring C_s , the concentration of free surfactants dissolved in

17 the aqueous phase of the process slurry.

18 Before describing the method used, it is useful to explain

19 that, for purposes of this specification, "free surfactants" is a term

20 used to denote the surface active compounds which are in aqueous solution

21 during hot water processing of a tar sand feed. It is believed that, in

22 the main, these compounds are the carboxylate salts which are the reaction

23 products of NaOH (the process aid) and carboxylic groups associated

24 with the bitumen.

25 It needs to be noted that not all of the carboxylates in the

26 aforesaid aqueous phase are surface active. Therefore, it is a necessary

27 part of the free surfactant determination method to differentiate between

28 the carboxylates or compounds which are surface active and those that are

29 not. This is further dealt with below.

1   The free surfactant determination procedure which has been

2 utilized takes advantage of the fact that surfactants collect at surfaces.

3 More particularly, a technique known as foam fractionation was used to

4 collect the surface active compounds present in a slurry aqueous phase

5 sample of known volume. This foam fractionation procedure is described

6 in the paper by C. Bowman entitled "Molecular and Interfacial Properties

7 of Athabasca Tar Sands", published in Proceedings 7th World Petroleum

8 Congress, $\underline{3}$, (1967), pages 583 - 604.

9   In greater detail, each sample of secondary tailings from

10 the batch extraction unit was centrifuged at 15000 G, to remove suspended

11 solids. A 200 cm^3 sample of the remaining aqueous phase was foam fraction-

12 ated in a 300 cm^3 cylindrical vessel equipped with a nitrogen sparger.

13 The introduction of gas was varied as required to yield a separable

14 foam. Fractionation was continued until the surface tension of the

15 residue reached a limiting value, substantially that of pure water, as

16 determined by the known maximum bubble pressure technique.

17   At this point, all of the surface active compounds were

18 isolated in the fractionate and the residue contained only non-surface

19 active compounds. The concentration of non-surface active compounds was

20 assumed to be equal in each of the fractionate and residue fractions.

21   50 cm^3 aliquots of each of the fractionate and residue

22 fractions were then titrated against hydrochloric acid, to establish

23 measures of the concentrations of the surface active and non-surface

24 active compounds.

25   Typical titration curves are illustrated in Figure 6. It

26 will be noted that the volumes of acid, used to titrate the non-surface

27 active carboxylates in the residue and the non-surface active plus surface

28 active carboxylates in the fractionate, can be determined from Figure 6

29 using the end points shown.

1   The data from these titrations were processed using the

2 following mathematical analysis to determine free surfactant concentration

3 (C_s), as follows:

4   - let C_{ns}^f be the concentration of non-surface active

5   carboxylates in the fractionate;

6   - let C_s^f be the concentration of surface active carboxylates

7   in the fractionate;

8   - let C_{ns}^r be the concentration of non-surface active

9   carboxylates in the residue;

10   - let C^f be the concentration of total carboxylates in the

11   fractionate;

12   - and let C^r be the concentration of total carboxylates in

13   the residue.

14   Therefore: $\quad C_{ns}^f + C_s^f = C^f \qquad\qquad$ (1)

15   and $\quad C_{ns}^r = C^r \qquad\qquad$ (2)

16   At equilibrium, the concentration of non-surface active

17 carboxylates is assumed to be the same in both the residue and

18 fractionate.

19 Therefore:

20     $C_{ns}^f = C_{ns}^r$

21 Thus, combining equations (1) and (2): $\quad C^r + C_s^f = C^f \qquad$ (3)

22   As C^f and C^r are determined by titration, the value of C_s^f

23 (the measure of free surfactant concentration) may be determined.

24   Detailed results of a free surfactant determination for

25 one marine ore feed D are now presented, in conjunction with Table I:

TABLE I

NaOH wt % on Feed	Tlgs vol cm^3	Volume frac'd cm^3	Vol of frac'te cm^3	Frac'te aliquot cm^3	Vol HCl cm^3	N_{HCl}	Vol resid cm^3	Resid aliquot cm^3	Vol HCl cm^3
.00	1080								
.04	1080	199.5	103.7	50.0	.49	.0571	91.3	50.0	.46
.08	1080	208.1	91.9	50.0	.48	.0571	115.4	50.0	.43
.12	1080	197.8	115.2	50.0	.89	.0571	79.7	50.0	.84
.20	1080	152.2	62.1	61.9	3.34	.0328	89.7	50.0	2.40

Total secondary tailings sample volume = 1080 cm^3

Fractionate carboxylate salt determination:

Total volume fractionated   = 152 cm^3 = V_{sample}

Volume of fractionate   = 62 cm^3 = $V_{fractionate}$

Aliquot volume   = 61.7 cm^3 = $V_{aliquot}$

Volume of HCl titrant   = 3.34 cm^3 = V_{HCl}

Normality of HCl   = 0.0328N = N_{HCl}

Therefore

Concentration of carboxylate salts in fractionate

$$= \frac{N_{HCl} \cdot V_{HCl}}{V_{aliquot}}$$

$$= 17.8 \times 10^{-4}$$

Residue carboxylate salt determination:

Residue volume   = 90 cm^3

Aliquot volume   = 50.0 cm^3 = $V_{aliquot}$

Volume of HCl titrant   = 2.40 cm^3 = V_{HCl}

Normality of HCl   = 0.0328N = N_{HCl}

1   Therefore

2   Concentration of carboxylate salts in residue

3
$$= \frac{N_{HCl} \cdot V_{HCl}}{V_{aliquot}}$$

5
$$= 15.7 \times 10^{-4}$$

6   From the equations

7    $C_s = c^f - c^r$

8    $= 17.8 \times 10^{-4} - 15.7 \times 10^{-4}$

9    $= 2.1 \times 10^{-4}$ equivalents per litre

10   This is the concentration of free surfactant in the 62.1 cm^3

11   fractionate sample. In the original tailings sample there is

12
$$\frac{(2.1 \times 10^{-4}) \times 62.1}{152}$$

13    $= 8.6 \times 10^{-5}$ equivalents per litre.

14   <u>Optimum Free Surfactant (C_o) Determination</u>

15   As previously stated, a number of hot water process extraction

16   runs were carried out on a particular feed in the batch extraction unit,

17   varying only the amount of NaOH addition. When the C_s values obtained

18   from the runs were plotted, a peak curve, such as one of the curves

19   shown in Figure 1, was obtained. This procedure was repeated for a

20   number of different feeds. It was discovered that the peaks of the

21   curves, corresponding with the maximum primary froth recoveries, fell

22   substantially on a vertical line corresponding with a single C_s value.

23   This particular optimum C_s value is referred to as C_o .

1   Stated otherwise, maximum primary froth recovery for the

2 various feeds occurs at only one small range of C_s values. Both below

3 and above that range, which for practical purposes is taken to be a

4 single value C_o , the primary froth recovery diminishes.

5   To summarize, for a given circuit or extraction unit, the

6 maximum primary froth recovery for various tar sand feeds always occurs

7 at substantially the same free surfactant concentration C_o in the aqueous

8 phase of the process slurry.

9 <u>The Linear Relationship (Figure 3)</u>

10   In conjunction with determining C_o for the extraction unit

11 used, one may determine, for each tar sand feed being considered, the

12 nature of the substantially linear relationship which exists between C_s

13 and NaOH addition.

14   This may be done by: extracting a plurality of portions of

15 each tar sand feed; using the hot water process at constant conditions

16 except for using different levels of NaOH addition; determining the C_s

17 value for each such extraction; and establishing the nature of the

18 linear relationship by plotting the linear surfactant production line for

19 each feed, which line expresses this relationship.

20   A typical plot of surfactant production lines, based on

21 experimental runs described below, is set forth in Figure 3. It will be

22 noted that each surfactant production line is extrapolated to intersect

23 the zero NaOH addition axis and provide an intercept value. Also, a

24 horizontal broken line is provided on the plot, which corresponds with

25 the C_o value for the extraction unit used.

26   Line A on Figure 3 is the surfactant production line obtained

27 in connection with extraction of a tar sand feed A, which is relatively

28 high in bitumen content. This feed had the following composition:

TABLE II

Tar Sand	Comments	Oil Content % w/w	Water Content % w/w	Solids Content % w/w	Fines Content (< -44) % w/w solids
A (rich)	fresh	13.7	1.5	84.8	8.9

The extraction procedure used on each tar sand sample, to develop the data for lines A (and line B described below),was as follows:

A charge of 500 g of the tar sand, 150 mL of water at 82°C, and different amounts of NaOH were introduced into the pot 1. Hot water was circulated through the jacket 2 to bring the charge to 82°C and to maintain it there. Once the charge was at temperature, it was agitated with the agitator 3 for 10 minutes at 600 rpm while simultaneously introducing air into the charge at 7 mL per second through the sparger 4. The air was then switched off and the mixture flooded with 900 mL of hot water (82°C). Mixing with the agitator was continued for a further 10 minutes. The agitator was then switched off. The produced primary froth was skimmed off the surface of the mixture and weighed. Samples thereof were analyzed. The residual mixture was then subjected to secondary separation. More particularly, it was agitated at 800 rpm for 5 minutes with air sparging at the rate of 4 mL/sec. The secondary froth produced was skimmed off, weighed and analyzed.

The extraction results for tar sand feed A were as follows:

TABLE III

NaOH % w/w tar sand	Mass Primary Froth (g)	Froth Composition (% w/w)			% Primary Recovery
		Oil	H_2O	Solids	
0.00	53.5	71.1	19.5	9.4	85.4
0.01	28.9	71.2	22.4	6.4	53.0
0.02	32.0	74.8	18.7	6.5	55.0
0.03	32.0	75.6	17.3	7.1	57.4

It will be noted from the composition set forth in Table II that line A is the surfactant production line for a tar sand feed high in bitumen content. As shown in Figure 3, the line obtained has a relatively steep slope, indicating that high concentrations of free surfactant are produced when extractions are carried out.

It will further be noted from Figure 3 , at zero NaOH addition, the bitumen-rich tar sand feed A produces such a high concentration of free surfactants that the C_s value is greater than the C_o value. The data of Table III indicates that the primary froth recoveries from processing this rich tar sand alone fall on the right hand downwardly sloping portion of the feed A curve on Figure 1. Thus, oil recovery by hot water extraction from this tar sand alone is less than the maximum possible, when this feed is processed in the ordinary way with NaOH addition.

Line B on Figure 3 is the surfactant production line obtained from extracting a tar sand feed B, known as marine ore, which was low in bitumen content and rich in clay particles. As shown, the slope of this line was less angular than that of line A, indicating that a lower concentration of free surfactants was generated.

Tar sand feed B had the following composition:

	TABLE IV				
Tar Sand	Comments	Oil Content % w/w	Water Content % w/w	Solids Content % w/w	Fines Content (< -44) % w/w solids
B (marine)	aged 60 days	5.6	10.3	85.0	21.0

The extraction results for tar sand feed B were as follows:

TABLE V

NaOH % w/w tar sand	Mass Primary Froth (g)	Froth Composition (% w/w)			% Primary Recovery
		Oil	H$_2$O	Solids	
0.01	1.01	26.35	64.64	9.01	3.6
0.03	1.20	36.45	54.88	8.67	4.3
0.06	1.40	42.72	49.97	7.31	4.1
0.12	1.32	38.66	52.94	8.40	4.7
0.16	1.99	47.58	44.98	7.44	7.1
0.20	1.51	40.27	51.52	8.22	5.4
0.24	0.84	39.83	52.16	8.01	3.0

As can be seen, the primary recovery of bitumen from this feed B was very low when it was processed alone.

Of particular interest is that portion of line B to the left of point 'a' on the plot. Here, for extractions conducted on feed B at NaOH additions in the range 0.0 to 0.04 wt %, there is a condition of free surfactant consumption. Stated otherwise, in the case of tar sand B and an NaOH addition in said range, the tar sand appears to be adsorbing the free surfactants that it produces when in the extraction slurry.

1   <u>Blending</u>

2   We postulated that, if a first tar sand feed, which is a

3   consumer of free surfactants, were to be blended with a second tar sand

4   feed, which is a producer of free surfactants, it might be possible to

5   achieve C_0 conditions. By 'producer' is meant that the tar sand, when

6   processed alone by the hot water process in accordance with conventional

7   conditions, will yield a surfactant production line which, in the context

8   of a Figure 3 plot, is always positive (as is the case with line A). By

9   'consumer' is meant that the tar sand yields a surfactant production

10   line which is partly negative (as is the case with line B).

11   In fact, it has been discovered that there is a synergistic

12   result obtainable, in that certain blends give higher recoveries than

13   would be expected by summing the results of processing each tar sand

14   feed separately.

15   This synergistic effect is shown in Figure 5. Feeds A and

16   B were blended in various proportions and subjected to bitumen extraction

17   in the previously described extraction unit. The extraction results were

18   as follows:

19   TABLE VI

20 21 22	Blend Composition (% Marine)	Maximum Primary Recovery (g bitumen/500 g feed)	Maximum Primary Recovery (%)
23	0	54	85
24	10	56	93
25	30	45	87
26	50	37	85
27	80	8	22
28	100	1	5

1   The straight line in Figure 5 is the calculated recovery that
2   was expected from blending alone, taking into account the proportion of
3   each feed in the blend. The curve depicts the actual recoveries obtained
4   when blends of tar sands A and B where extracted as described above. The
5   upper part of the curve, between the left axis of the plot and the node
6   point, shows enhanced recoveries due to synergism.

7   The node point on Figure 5 corresponds with point 'a' on
8   Figure 3.

9   The shaded area on Figure 3 defines the blend proportions
10   which will yield synergism.

11   Figure 4 is a plot showing the surfactant production lines
12   for two tar sand feeds C and D to be blended, where the left hand end
13   of the line for the producer feed is below the C_o line when small NaOH
14   additions are used in the extractions. Only when enough NaOH is used, so
15   as to bring the producer line to at least C_o , can synergism result. So,
16   for the blend of Figure 4, the shaded area is shifted to the right in
17   comparison to that of Figure 3.

18   One may select compatible tar sand feeds to make up a
19   blended feedstock which is amenable to processing at C_o condition, by
20   observing the following rules:

21   (1) select a first tar sand feed whose surfactant
22   production line has a negative intercept value when
23   plotted on a plot of the type of Figures 3 and 4 (that
24   is , a feed which will consume free surfactants in the
25   course of hot water extraction);
26   (2) select a second tar sand feed whose surfactant
27   production line has a positive intercept value and
28   which extends above the C_o line (that is, a feed which
29   will produce free surfactants in the course of hot
30   water extraction in the absence of NaOH);

(3) the first feed surfactant production line having to cross
the zero free surfactant concentration axis at a NaOH
addition value which is less than 0.2 wt. % and greater
than

    (a) the NaOH addition value corresponding with the
        second feed intercept (in which case the value is 0)

                  or

    (b) the NaOH addition value corresponding with the point
        at which the second feed surfactant production line
        crosses the C_o line,

whichever is greater. Stated otherwise, the first
vertical boundary line, passing through the point where
the first feed surfactant production line crosses the
zero free surfactant concentration axis, is to the right
of or at a greater NaOH value than the second vertical
boundary line, which passes through the point where the
second feed surfactant production line crosses the C_o
line or, if it does not so cross, which coincides with
the zero NaOH addition axis.

The amount of NaOH used in conjunction with the blended
feedstock is selected so as to fall within the range of values between
the two vertical boundary lines.

Having selected the feeds and NaOH addition to be used, one
may determine the proportions of each feed to be used by solving the
following equations:

$$C_o = X_1 (C_{s,1} + R_1 P) + X_2 (C_{s,2} + R_2 P) \qquad (4)$$

$$1 = X_1 + X_2 \qquad\qquad\qquad\qquad\qquad (5)$$

where:

1 C_o is the optimum concentration of free surfactant

2 in the aqueous phase of the process slurry for the

3 extraction unit used

4 $C_{s,1}$ and $C_{s,2}$ are the intercepts for the selected first

5 and second feeds

6 R_1 and R_2 are the slopes of the free surfactant production

7 lines for the first and second feeds

8 P is the process aid addition selected

9 X_1 and X_2 are the proportions of first and second feeds

10 used to make the blend.

11 There may, of course, be more than two components in the

12 blend, in which case all values of X will give a sum of 1.

13 The improvement in primary froth recovery which can be

14 obtained by selecting free surfactant consuming and producing tar sand

15 feeds as set forth, combining them with a NaOH addition selected from the

16 range between the vertical boundary lines, and combining the tar sands in

17 proportions established by solving equations (4) and (5),is demonstrated

18 by the following example involving a blend of feeds A and B.

19 Inspection of Figure 2 shows that C_o for the extraction

20 unit of Figure 1 was 1.2 and 10^{-4} N (i.e. equivalents per litre).

21 Turning to Figure 3, $C_{s,a}$ for feed A at zero NaOH addition was 1.45 x

22 10^{-4} N. The slope R_a of the free surfactant line for feed A was 44.8 x

23 10^{-4} . For feed B, $C_{s,b}$ was -0.45 x 10^{-4} N and R_b was 11.9 x 10^{-4} .

24 With this blend, the left hand vertical boundary line coincided

25 with the zero NaOH addition axis, and the right hand vertical boundary

26 line occurred at 0.04 weight % NaOH. That is, the amount of NaOH to be

27 added fell between 0.0 and 0.04 wt. %. With the aid of equations (4)

28 and (5) , one can calculate the amount of NaOH to be added for any blend

29 of these feeds, to give maximum recovery.

1   For example, one can select a blend having a NaOH addition

2   of 0.01%. Solving equations (4) and (5) , one obtains recommended pro-

3   portions of feed A and feed B as follows:

4        $X_1 = 0.69$

5        $X_2 = 0.31$

6   Hence one should blend

7   69% feed A with

8   31% feed B,

9   when one uses a process aid level of 0.01 wt. % NaOH.

10   In fact, we blended 70% feed A and 30% feed B and performed

11   extraction experiments at various NaOH levels from 0.0 to 0.04 wt. %.

12   We then interpolated the results to find the bitumen in primary froth

13   at the 0.01 wt. % NaOH level. We found the result to be 45 g bitumen.

14   This is very close to the value of 46 g for a 70/30 blend (Figure 5).

15   It remains to show that an improvement is obtained by pro-

16   cessing the blend, compared with processing each feed separately, at

17   the respective C_o conditions.

18   Feed A, processed at 0.0 wt. % NaOH, gave 53.5 g bitumen.

19   (0.0 NaOH is the nearest approach to C_o for this feed [Figure 3]. The

20   recovery was 85.4% of the maximum attainable [Figure 2]. Feed B was

21   processed at 0.16% NaOH [this being as close as we came to the ideal

22   value of 0.14, in our experimental program] .) It gave 2.0 g bitumen,

23   which, according to Figure 2, was equivalent to 7.1 % of the maximum

24   attainable.

25   Hence:

26   Feed   Recovery

27   250 g Feed A

28   + 250 g Feed B

29   processed separately 0.7 x 53.5 + 0.3 x 2.0 = 38 g

30   250 g Feed A

31   blended with

32   250 g Feed B   45.0 g

1   These points can also be read off Figure 5. As can be seen, there was an

2   18% improvement in recovery as a result of blending. It was known that

3   the value of C_0 was reached in this case from equations (4) and (5)

4   set forth above.

1 THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE

2 PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

3 1. A process for extracting bitumen from tar sand of

4 varying nature using the hot water process in an extraction circuit,

5 wherein the tar sand is conditioned, by slurring it with hot water and

6 alkaline process aid with agitation, is diluted with water, and is then

7 retained in a quiescent condition to produce primary bitumen froth, the

8 improvement comprising:

9 selecting a first tar sand feed which, when slurried, is a

10 consumer of free surfactants and a second tar sand feed which, when slurried,

11 is a producer of free surfactants;

12 and blending said first and second tar sand feeds and process

13 aid in the conditioning step in amounts selected to yield substantially

14 the optimum free surfactant concentration, in the aqueous phase of the

15 process slurry for the circuit, required to yield maximum primary froth

16 recovery.

17 2. A process for extracting bitumen from tar sand of

18 varying nature using the hot water process in an extraction circuit,

19 wherein the tar sand is conditioned, by slurrying it with hot water and

20 alkaline process aid with agitation, is diluted with water, and is then

21 retained in a quiescent condition to produce primary bitumen froth, the

22 improvement comprising:

23 determining, for the circuit involved, a measure of the

24 optimum concentration of free surfactant in the aqueous phase of the

25 process slurry, which substantially corresponds with maximum primary

26 bitumen froth recovery from that circuit;

27 selecting a first tar sand feed which, when slurried, is

28 a consumer of free surfactants and a second tar sand feed which, when slurried,

29 is a producer of free surfactants;

1   and blending said first and second tar sand feeds and process

2   aid in the conditioning step in amounts selected to yield substantially the

3   optimum free surfactant concentration, in the aqueous phase of the process

4   slurry for the circuit, required to yield maximum primary froth recovery.

5   3. In the process of extracting bitumen from tar sand of

6   varying nature using the hot water process in an extraction circuit,

7   wherein the tar sand is slurried in a conditioning drum with hot water

8   and alkaline process aid, agitated, and then retained in a quiescent

9   condition to produce primary bitumen froth, the improvement comprising:

10   (a) determining, for the circuit involved, a measure of the

11   optimum concentration of free surfactant in the aqueous phase of the

12   process slurry, which substantially corresponds with maximum primary

13   bitumen froth recovery from that circuit;

14   (b) determining, for each of a plurality of tar sand feeds

15   which are proposed for use to make a blended feedstock, the substantially

16   linear relationship which exists between a measure of the concentrations

17   of free surfactant, present in the aqueous phase of its process slurries,

18   and the amounts of process aid added, when a plurality of hot water pro-

19   cess extractions are practised on each tar sand feed at different levels

20   of process aid addition, all other conditions being maintained substantial-

21   ly constant;

22   (c) selecting first and second tar sand feeds, from the

23   group tested, such that, if the test data from steps (a) and (b) were

24   plotted on a graph and cross-hatched in accordance with the appropriate

25   figure selected from the plot of free surfactant concentration in the

26   aqueous phase of the process slurry against the amount of process aid

27   added, the first feed would have a surfactant production line whose

28   intercept is above the zero surfactant concentration line and would have

29   values above the optimum surfactant concentration line at a

30   process aid addition value of less than 0.2 wt. %, the second feed

31   would have a surfactant production line whose intercept is below the

1   zero surfactant concentration line and would cross the zero surfactant

2   concentration line at a process aid addition value less than 0.2 wt. %

3   and greater than the process aid value corresponding with either the

4   first feed intercept or the point where the first feed surfactant pro-

5   duction line crosses the optimum surfactant concentration line, which-

6   ever is greater;

7   (d) selecting the process aid addition from the range of

8   values between the vertical boundaries of the cross-hatched area;

9   (e) determining the proportions of first and second tar sand

10   feeds to make a blended feedstock so that they satisfy the following

11   equations:

12
$$C_o = X_1 (C_{s,1} + R_1 P) + X_2 (C_{s,2} + R_2 P)$$

13
$$1 = X_1 + X_2$$

14   where: C_o is the value determined in Step (a)

15         $C_{s,1}$ and $C_{s,2}$ are the intercepts for the selected

16   first and second feeds

17   R_1 and R_2 are the slopes of the surfactant production

18   lines for said first and second feeds

19   P   is the process aid addition selected

20   X_1 and X_2 are the proportions of first and second

21   feeds used to make the blend;

22   (f) and supplying to the extraction process said first and

23   second tar sand feeds and process aid in accordance with the values for

24   X_1, X_2 and P.

- 29 -

Fig.1.

LABORATORY BATCH EXTRACTION UNIT
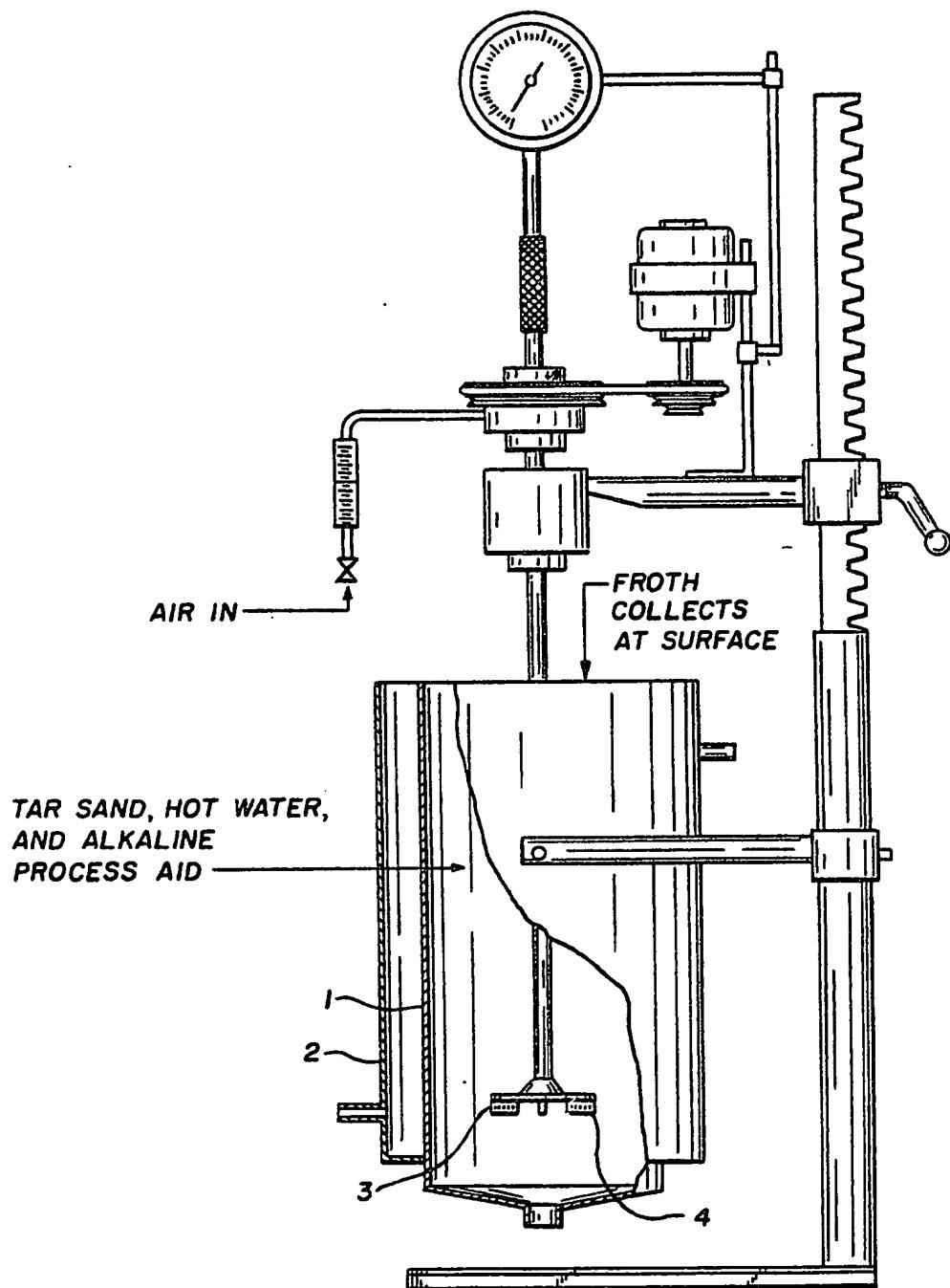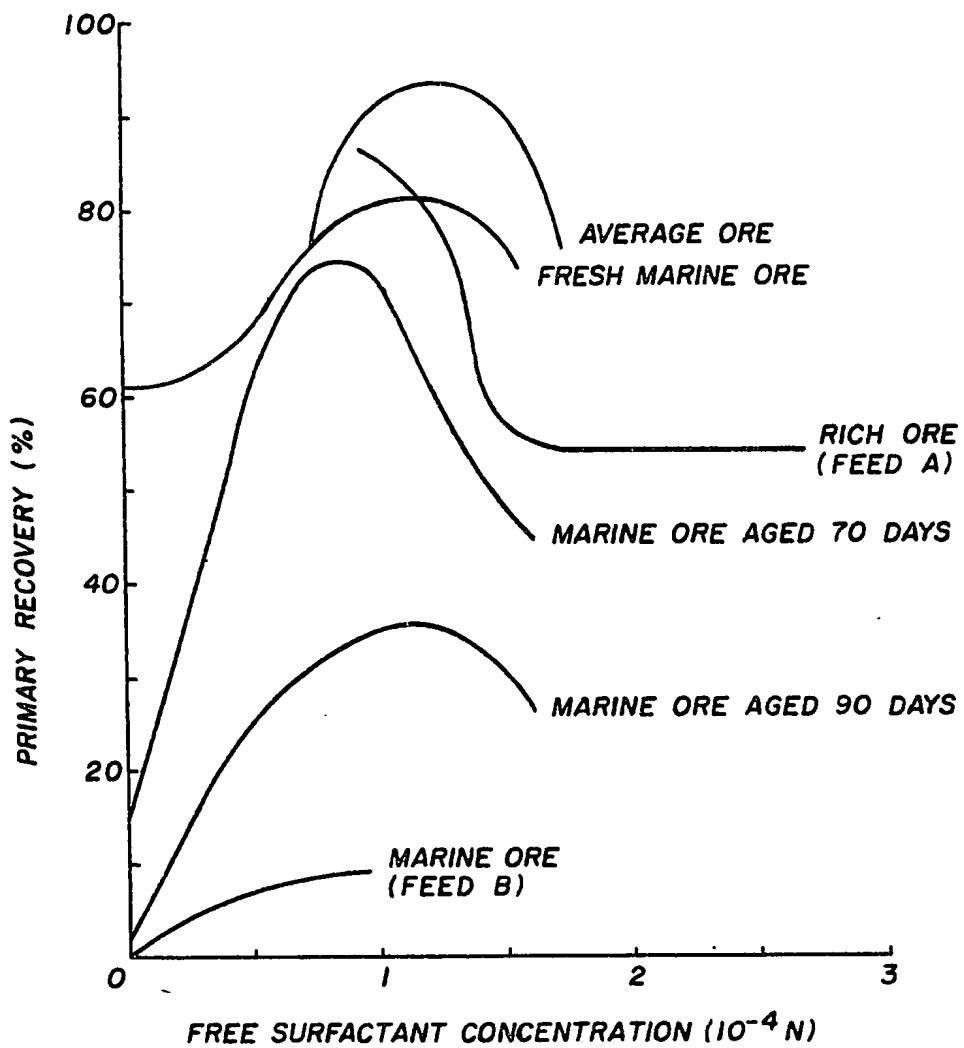
AIR IN

FROTH
COLLECTS
AT SURFACE

TAR SAND, HOT WATER,
AND ALKALINE
PROCESS AID

1

2

3

4

Patent agent:
EP Johnson

Fig.2.

Plot with y-axis labeled "PRIMARY RECOVERY (%)" ranging from 0 to 100, and x-axis labeled "FREE SURFACTANT CONCENTRATION (10⁻⁴ N)" ranging from 0 to 3. Curves labeled: AVERAGE ORE, FRESH MARINE ORE, RICH ORE (FEED A), MARINE ORE AGED 70 DAYS, MARINE ORE AGED 90 DAYS, MARINE ORE (FEED B).

Patent agent:
EP Johnson

Fig 3.

Fig.4.

1214421

6 - 4
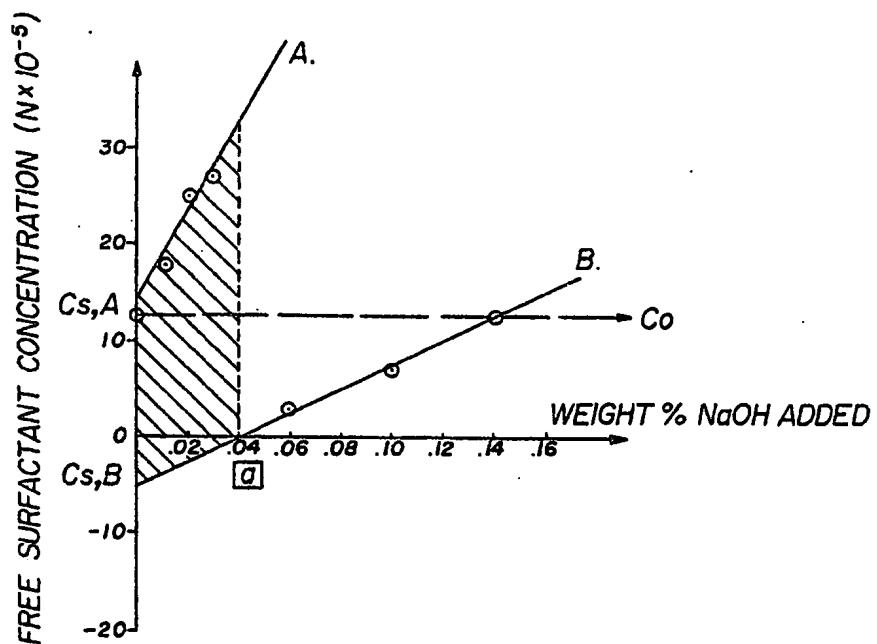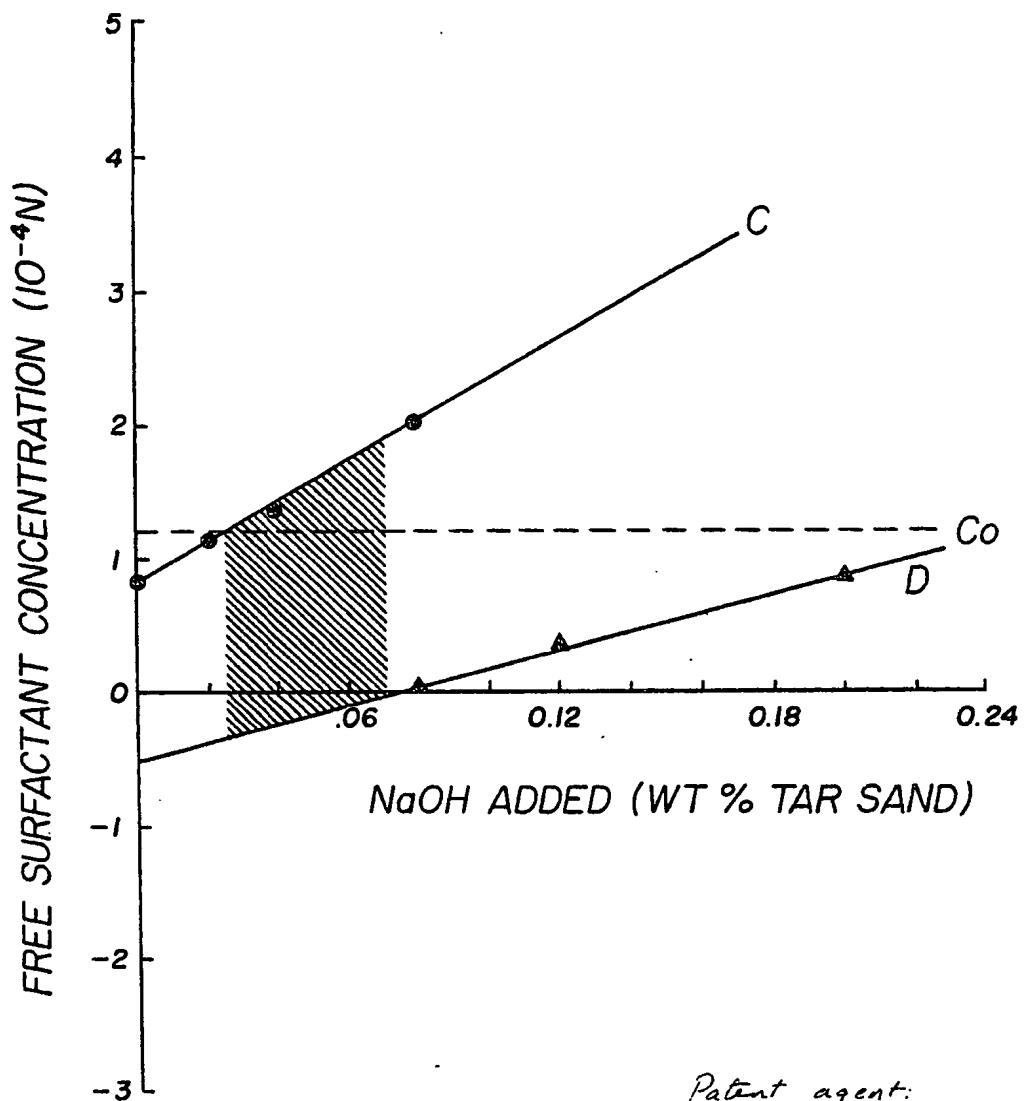
Fig. 5.

MAXIMUM PRIMARY RECOVERY
VS. % MARINE ORE IN BLEND

NODE POINT ⟶

BITUMEN IN PRIMARY FROTH $\left\{ \frac{g \text{ BITUMEN PER}}{500 g \text{ TAR SAND}} \right\}$

WEIGHT % MARINE ORE IN BLEND
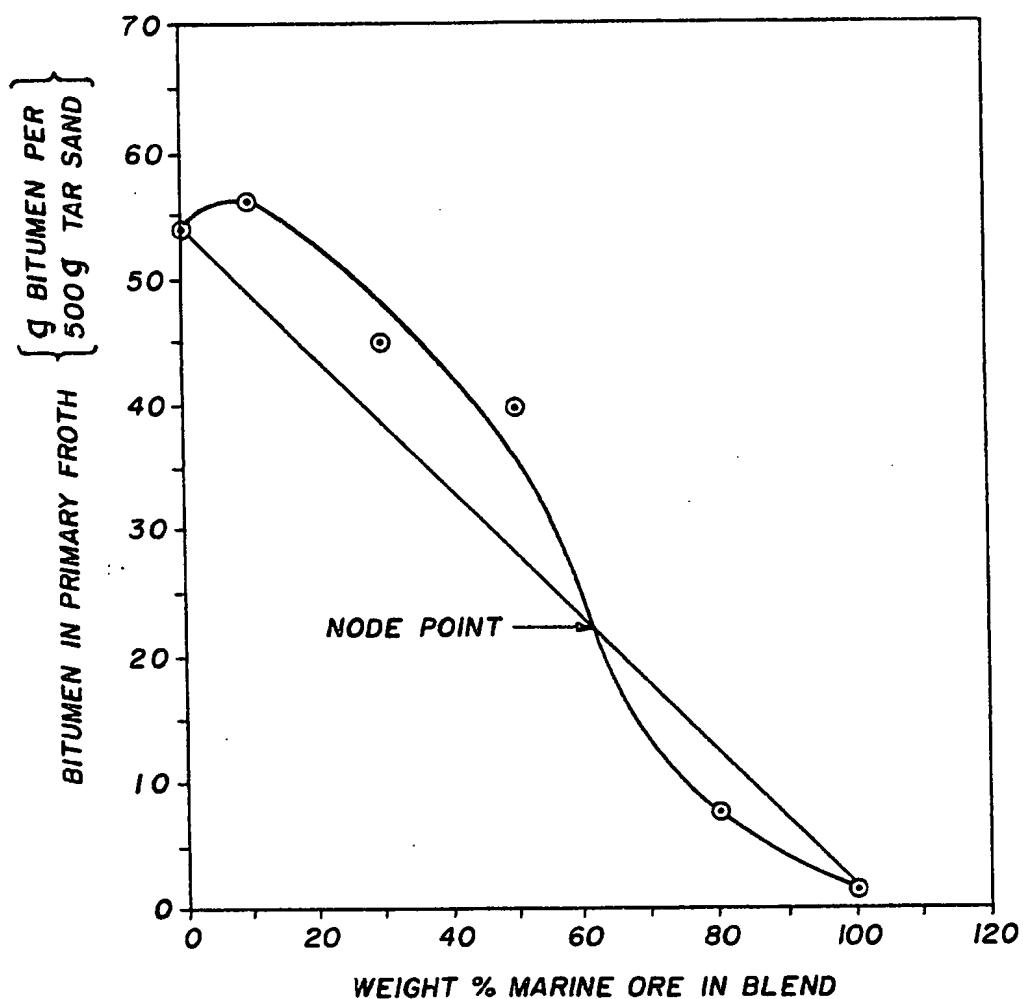
Patent agent:
RP Johnson

Fig.6.

ACID TITRANT VERSUS pH
TO DETERMINE SURFACTANT
CONCENTRATION

RESIDUE

SODIUM HYDROXIDE
END POINT

SODIUM CARBOXYLATE
END POINT

VOLUME 0.038N HYDROCHLORIC ACID (ml)

FRACTIONATE

SODIUM HYDROXIDE
END POINT

SODIUM CARBOXYLATE
END POINT

VOLUME 0.038N HYDROCHLORIC ACID (ml)

Patent agent:
EP Johnson